Microstructural evolution and thermal stability after aging of a cobalt-containing martensitic bearing steel

Shaohong Li [a],*, Kunyu Zhao [a], Kang Wang [a], Maosheng Yang [b]

[a] School of Materials Science and Engineering, Kunming University of Science and Technology, Kunming 650093, China
[b] Department of Structural Materials, Central Iron and Steel Research Institute, Beijing 100081, China

ARTICLE INFO

ABSTRACT

The microstructural changes and thermal stability of a cobalt-containing martensitic heat resistance bearing steel have been investigated in this paper. The hardness variation showed a progressive hardness decrease associated with coarsening of fine carbides at elevated temperatures. The precipitation of secondary phases during tempering at 500 °C for 10 h and 100 h has been characterized and identified in detail using transmission electron microscopy. The results revealed that the aging treatment induced very fine secondary M_6C precipitates which were responsible for the secondary hardening peak when tempered at 500 °C for 30 h. But the hardness gradually decreased due to the coarsening of M_6C carbide and other secondary phases (such as µ phase, σ phase, and χ phase) precipitation when the samples were tempered over 30 h at 500 °C.

© 2016 Elsevier Inc. All rights reserved.

1. Introduction

Nowadays, the progress in the bearing industry has increased the requirements of high performance bearing steels with demanding service environment. Conventional hardened bearing steels such as GCr15, AISI 52100 and M-50 cannot meet these requirements [1] in advanced aerospace engines, wind turbines, and high-speed railways, etc. The increased service temperature and the longer service life of bearing requirements have accelerated the development of high-performance bearing steels. Therefore, several varieties of bearing steels have been developed to meet the increasingly stringent requirements for high power density and reliability of bearings for applications [2]

The microstructure is one of the most significant factors that governs the mechanical properties and service performance of materials. In addition, the stability of the microstructure after heat treatment and during service is one of the fundamental requirements concerning the bearing industrial applications. Generally, in single-phase alloys, microstructure refinement can improve strength. However, if second phase particles are produced in the matrix, the particle size, fraction, distribution and the morphology play an important role in strengthening of materials [3]. Therefore, the microstructure in high strength bearing steels is believed to evolve during service under extreme operation conditions, such as under alternating stress and/or the high-temperature environment. Since the microstructure changes during service, decay in mechanical properties may lead to components failure and carbide

stability is reported to strongly influence bearing performance [4]. During operation under heavy loads and high temperature, the bearings were subjected to several microstructural changes. Hence the thermal stability of microstructure of bearing steel plays a very important role for the practical applications at elevated temperatures. It was suggested that microstructure stability can be improved by various alloying elements or composite reinforcements and thermal stability depends on many variables, such as stacking fault energy of the material, processing or properties of grain boundaries [5]. The authors have pointed out that a major aim in controlling bearing steel microstructures is to maximize their resistance to rolling contact fatigue [6–8]. Precipitates dissolution is the main microstructural change associated with rolling contact fatigue and precipitates stability is paramount in enhancing bearing life [9].

Cryogenic treatment is a supplementary process to conventional heat treatment process in steels which have been proved to be an effective technique enhancing abrasive wear performance [10], dimension stability [11] and fatigue life improvement [12,13]. Barron [14] has found that cryogenic treatment can improve the wear resistance of SAE 52100 bearing steel. Harish et al. [15] have investigated the microstructural changes of cryogenically treated En 31 bearing steel and concluded that the cryogenic treatment should be followed by tempering to promote secondary carbide precipitation which is essential for hardness augmentation and wear resistance improvement.

The formation of precipitates during aging plays a very important role on mechanical properties of martensitic steel. It is important to study the microstructural evolution of high performance bearing steels during heat treatment so that a better understanding can be obtained on how to control the microstructure for the long-term service. The

---

* Corresponding author at: 253# Xufu Rd., Wuhua District, Kunming City, Yunnan Province, China.
  E-mail address: uniquelsh@163.com (S. Li).

main objective of this study is to investigate the microstructure changes and propose an explanation for the phenomena of variation of hardness during service at high temperatures of a newly developed bearing steel after cryogenic treatment.

2. Material and Experimental Methods

The tested steel is produced in a vacuum induction melting furnace and the ingots are forged into ⌀20 mm bars. The chemical compositions of the studied steel are: 0.15%C, 0.06%Si, 14.0%Cr, 13.0%Co, 4.5%Mo, 2.0%Ni and balance Fe (in wt.%). Heat treatment cycles included austenitization are carried out at 1020– 1050 °C for 1 h in vacuum furnace before quenching into oil. The samples are put into a refrigerator at −75 °C for 2 h after quenching and warm up to room temperature in the air. And then the samples are tempered at 480– 510 °C for 2 h. The detailed treatment processes of the steel are listed in Table 1.

For all hardness measurement, Rockwell hardness tester was used. A load of 150 kg for 30 s was applied on all samples to measure the hardness. Each effective hardness value of the sample is the average hardness of five points. The impact toughness is determined for Charpy V-notched specimens 10 mm × 10 mm × 55 mm in size. The volume fraction of retained austenite in the steel is determined by D/max-2200 X-ray diffraction with a scan step of 0.05° per second. The microstructure was etched by a solution consisting of 50 ml HCl, 2 ml HNO_3, 1 g $CuCl_2$, 2.5 g $FeCl_3$, 50 ml alcohol and 50 ml H_2O and evaluated by Leica DMILM optical microscope (OM), HITACHI S-4300 scanning electron microscope (SEM). The morphology and the type of secondary phases in different treatments have been identified by the JEM-2100 transmission electron microscope (TEM).

3. Experimental Results and Discussion

3.1. Microstructure Characterization of Cryogenic Treated and Tempered Samples

Quenching and tempering is a common process used in the manufacture of steel components. During quenching and tempering process, as-quenched steels are tempered to achieve optimum mechanical properties tailored to fit a specific application and the final properties of a quenched and tempered steel part depends on the microstructure evolution during the process [16].

Fig. 1 shows the microstructures of the samples austenitized at different temperatures from 1020– 1050 °C and then cryogenic treated at −75 °C for 2 h. It is obviously that the microstructure is mainly martensite matrix. However, the δ-ferrite begins to produce while the austenitizing temperature elevates to 1040 °C. Liu et al. [17] pointed out that the δ-ferrite produce easily when austenitized at a higher temperature because the ferrite stabilizing elements such as Mo and Cr increased the chromium equivalent (Cr_{eq}) of the steel. The δ-ferrite is a relatively soft phase in the martensite and it reduces the strength of the steel, increases its ductility but not always its fracture toughness [18]. The microstructures of the samples tempered at 510 °C after quenching at different temperatures and then cryogenic treated at −75 °C for 2 h are presented in Fig. 2. It is clearly that the martensitic laths are coarsened while the sample was carried out at higher austenitizing temperature. This is attributed to the increase of original austenite grain size with the elevated austenitizing temperature.

In order to identify the phases in the microstructure after different treatments mentioned above, the X-ray diffraction technique is used to determine the crystalline structure and measure the volume fraction of retained austenite and martensite at room temperature using Cu Ka ($\lambda = 1.5418 \times 10^{-10}$ m) radiation with vanadium filters. The volume fraction of retained austenite was estimated in accordance with ASTM standard E975-00 standard [19], considering the diffraction crystal plane (200), (220), (311) of retained austenite and (200), (211), (220) of martensite. Fig. 3 gives the X-ray diffraction patterns of the tested steel before and after cryogenic treatment. The peaks corresponding to retained austenite and martensite in the X-ray diffraction pattern graph are available. The austenitic reflection (111) A revealed as a shoulder at the martensitic doublet (110) M. The austenite diffraction peaks of (200) A, (220) A and (311) A crystal plane are still notable after tempering as is shown in the graph. As labeled in Fig. 3(a) and (b), there are a large amount of unstable retained austenite (about 17%) which remains in the specimen after quenching. However, according to the XRD results in Fig. 3(b), it can be seen clearly that the peaks of retained austenite exhibit a low-intensity value after cryogenic treatment, which validates that the retained austenite transformed into martensite during cryogenic treatment. The measured phase volume fractions of retained austenite before and after cryogenic treatment are 17% and 4.1% respectively. The scattered intensities of the retained austenite and martensite reflections after quenching at 1020– 1050 °C and then cryogenic treated at −75 °C for 2 h are shown as a function of the scattering angle 2θ in Fig. 3(c). The volume fractions of the retained austenite of quenching at 1020– 1050 °C after −75 °C cryogenic treatment are under 5%. Authors [20,21] have suggested that cryogenic treatment carried out after quenching and before tempering, almost completely converts the retained austenite in AISI D2 steel to martensite. However, Li et al. [22] have pointed out that the retained austenite transformed into martensite during cryogenic treatment but not completely and presented in a thin film between the laths of martensite and stably exists even during prolonged soaking time in liquid nitrogen. As given in Fig. 4, the retained austenite had not been transformed into martensite completely even carrying out at high-temperature tempering. Authors [23,24] have declared that austenite can often be retained in two forms: blocky and film types. The former is unstable, which significantly influences the dimensional stability of the material [25]. The film austenite, located between martensite plates, is very fine and stable due to the carbon enrichment. As is known, that aging or tempering of quenched steel can make the retained austenite stabilized [26,27], including thermal and chemical stability. The carbon atoms initially diffuse primarily from martensite to the martensite/austenite interfaces and segregate there, at later stages the diffusion of carbon atoms into austenite takes place resulting in enrichment of carbon in austenite, thereby the enriched carbon retained austenite keeps stable during subsequent cooling to ambient temperature [28]. Authors [29] have proposed that the interstitial carbon atoms can also migrate to the defects

Table 1
Detailed treatment processes of the steel.

Heat treatment	Quenching temperature (Q)	1st cryo-treatment (QC)	1st tempering (QCT)	2nd cryo-treatment (QCTC)	2nd tempering (QCTCT)
A	1020 °C × 1 h	−75 °C × 2 h	–	–	–
B	1030 °C × 1 h	−75 °C × 2 h	–	–	–
C	1040 °C × 1 h	−75 °C × 2 h	–	–	–
D	1050 °C × 1 h	−75 °C × 2 h	–	–	–
E	1050 °C × 1 h	−75 °C × 2 h	510 °C × 2 h	–	–
F	1050 °C × 1 h	−75 °C × 2 h	510 °C × 2 h	-75 °C × 2 h	–
G	1050 °C × 1 h	−75 °C × 2 h	510 °C × 2 h	-75 °C × 2 h	510 °C × 2 h